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SOVIET RESEARCH IN POWDER METAL MATERIALS

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SOVIET RESEARCH IN POWDER METAL MATERIALS

This publication contains the translations of six articles from the Russian-language periodical, Metallokeramicheskiye materialy i metody ikh issledovaniya, -- Informatsionnyye materialy (Powder Metal Materials and Methods of Investigating Them -- Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev, 1959. Complete bibliographic information accompanies each article.

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SINTERING CHROMIUM BORIDE

/Following is a translation of an article by P.S. Kislyy and V.S. Neshpor, in the Russian-language periodical Metallokeramicheskiye Materialy i Metody ikh Issledovaniya -Informatsionnyye Materialy (Powder Metal Materials and Methods of Investigating Them -Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev 1959, pages 11-12./

Chromium boride (CrB_2) is a hard, high-melting material with metallic properties. The extreme hardness and stability of chromium boride, in combination with its resistance to corrosion by gas and its relatively high thermal stability (a cylindrical sample of CrB_2 with diameter 8 mm and length 21mm will withstand 40 thermal changes without shattering), permits using chromium boride as a refractory material and also in heat-durable and resistant alloys.

Usually, chromium boride is obtained in the form of a powder, during the reduction of chromium oxide by boride carbide in a vacuum, or by direct synthesis from components in the hard state. Research has been done on the technolog-

ical conditions for converting chromium boride powder into the compact state by methods of powder metallurgy.

Heat Pressing

The heat pressing of chromium boride powder is effected in a laboratory press, in graphite press-molds /1/.

Compact cylindrical forms with diameter 8-10 mm and length 10-20 mm, with minimal residual porosity (less than 3%), were obtained by submitting powder to a pressure of 180 kilograms per square centimeter and to a sintering temperature of 2000±50° C. Sintering occurred in 10-12 minutes. The forms obtained had a fine-grained, etched cross-section with a metallic glitter, without pores and defects noticeable to the eye. For easier separation of the forms from the press-mold, the latter, after the powder was poured into it, was greased with a thin mixture of glycerin and flaked graphite. Experience showed that, in the heat pressing of chromium boride powder, it was not necessary to use a special protective atmosphere - the carbon monoxide, formed during the partial scorching of the press-mold, is sufficient to protect the material from oxidation. The sintered chromium boride contained not more than 1.03% of total carbon, when the original powder contained 0.91% carbon.

Usual Pressing and Sintering

Samples of chromium boride, of rectangular shape, were pressed in a steel, detached press-mold with the addition, as a binding, of a 5% solution of rubber in benzine, under a pressure of 3-5 tons/square centimeter. Sintering of the pressed samples took place in a carbon-tubular stove, in a stream of hydrogen. The samples for sintering were placed on graphite blocks, lubricated with flaked graphite, and were covered on top with graphite plates to guarantee equal heating and the prevention of unequal sintering and distortion as a consequence of a temperature gradient. Compact forms with a residual porosity of not more than 4-8% were obtained at a temperature of sintering of 2100-2150°, in the course of 15-20 minutes.

Various properties of compact chromium boride were studied.

The fusion temperature of chromium boride, which turned out to equal 2200±50°C, was determined according to a method described in operation [2].

The limit of durability for compression, established for maximum loading, which the samples withstood without destruction, during their compression in a hydraulic press,

amounted to 60-70 kilograms/square millimeter (according to data found in literature, the limit of durability of CrB_2, for tension and bending, amounts to, correspondingly, 25 and 29 kilograms/square millimeter).

The coefficient of thermal expansion of CrB_2, determined dilatometrically, amounts to $11.6 \cdot 10^{6}$ degrees^{-1} (during porosity of the samples of 2-3%).

The corrosion resistance of the samples of chromium boride (porosity 6%), at 1200 C, was determined by the method of continuous weighing.

After a 23-hour exposure at this temperature, the sample increased in weight by 0.021%, in total. The constant of the speed of corrosion for chromium boride is $6.5 \cdot 10^{-10}$ grams/square centimeter min., which is on the order of 1-2 less than for borides of titanium and niobium /3/.

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EQUIPMENT FOR DETERMINING THE KINETICS OF VAPORIZATION AND THE VAPOR TENSION OF METAL POWDERS

W, Mo

Following is a translation of an article by I.M. Fedorchenko and Yu.B. Ermolovich, in the Russian-language periodical Metallokeramicheskiye Materialy i Metody Ikh Issledovaniya —Informatsionnyye Materialy (Powder Metal Materials and Methods of Investigating Them —Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev 1959, pages 13-16.

A method for determining the kinetics of vaporization and the vapor tension of metal powders has been developed in the Institute of Powder Metals and Special Alloys of the Academy of Sciences, USSR. Below, is a description of the experimental equipment for realizing this method.

An outline of the internal arrangement of the vacuum chamber is shown in Figure 1. Vaporization occurs from the plane surface of the heater 3, which is a band made out of tungsten or molybdenum, heated by the passage of an electric current going into the vacuum chamber along

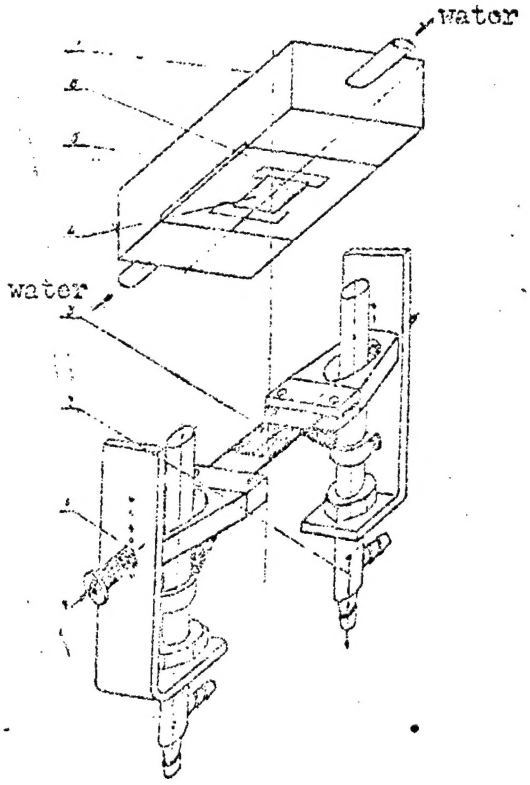

Figure 1

Outline of the Internal Arrangement

the current carriers 2, cooled by running water. The surface of the band, on which the metal powder is placed in a thin layer, has the dimensions 2x2 square centimeters.

The band is heated by the passage of a stabilized current from a stabilizer of 5 kilowatts, which provides a continuous and even temperature for the whole area of

vaporization, which remains flat and horizontal during the heating process thanks to the use of a tension spring 1.

The temperature is measured by a platinum-platinum-rhodium or chromel-alumel thermocouple; temperature control is also possible by a optical pyrometer, effected through a window in the vacuum chamber.

The vaporized atoms condense on the glass plate, with the vapor in the vacuum with the copper electrodes 5, which is first deposited on it. The places of continuous contact of the measuring circuit with the dust covered electrodes, which must have considerably less resistance than the condensate being studied, are shown by pointers in Figure 1. The dimensions of the section on which condensations occurs, that is, of the condenser also, are 2x2 square centimeters (cross-hatched in Figure 1), which is provided by the use of a special diaphragm, placed in front of the condenser (not shown in Figure 1). The glass plate is tightly pressed against the polished wall of the copper block 7, cooled by running water.

The external view of the vacuum chamber, together with the diffusion pump, is shown in Figure 2. The vacuum inside it is created by the action of a Faure-vacuum pump PVN-20 and a diffusion pump TsVL-100 and attains $2 \cdot 10^{-6}$ mm Hg. The

system of two valves and two pumps, RVN-20, provides the possibility of emptying and filling the chamber without cooling of the diffusion pump, which decreases time between experiments up to 20 ÷ 30 minutes. In the chamber, there is also a window and three mobile connections, which permits various manipulations inside it. Thanks to the connection of the evacuating system to the side wall of the chamber, it was possible to introduce 8 leads into the chamber, through the bottom of it, to which are connected the ends of the thermocouple, the circuits for measuring the electrical conductivity of the condensate, for the heating joint and the block, which is cooled by water. There are apertures for the connection of the lamps of the vacuum gage, in the lids of the chamber and diffusion pump. The walls of the chamber are cooled by running water. Operation of the equipment consists of the following stages:

1. Dusting of the copper electrodes on a specially cut out and cleaned glass plate. During this, copper powder is placed in a thin layer on the molybdenum band of the vaporizer, which is heated in the vacuum at a temperature of 1000 C. The obtaining of the necessary dimensions of electrodes is made possible by the use of a special diaphragm, placed in front of the glass plate.

2. The application of a layer of the metal being studied of such a thickness, beginning from which the electrical conductivity of the film is already proportional to its thickness. The size of the layer is controlled by measuring its electrical conductivity.

3. The experiment proper.

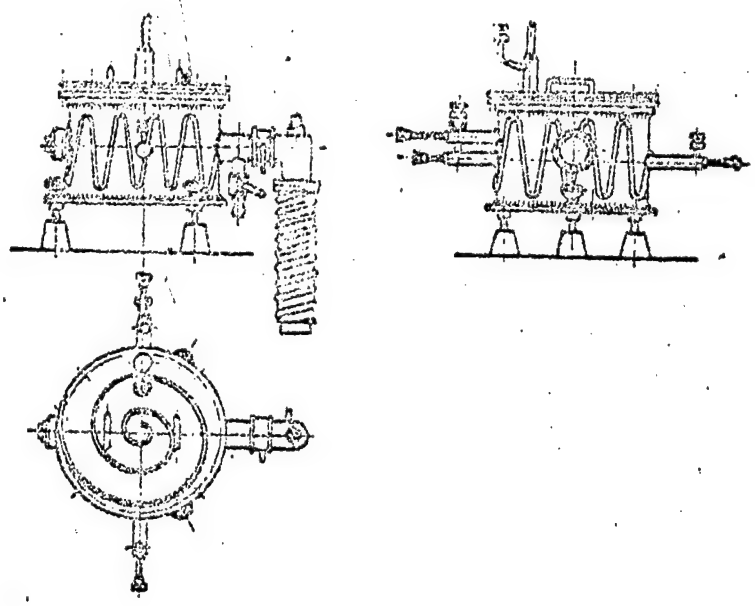

Figure 2

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METHODS OF PREPARING ALLOYS OF TITANIUM CARBIDE WITH MOLYBDENUM

Mo

Following is a translation of an article by V.N. Eremenko and T.Ya. Velikanova, in the Russian-language periodical Metallokeramicheskiye Materialy i Metody Ikh Issledovaniya - Informatsionnyye Materialy (Powder Metal Materials and Methods of Investigating Them--Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev 1959, pages 25-26.

As our many experiments have demonstrated, the obtaining of uniform alloys of titanium carbide with molybdenum does not appear to be possible because of sharp liquation according to specific gravity. The experiments were conducted in an arc furnace with a copper, cooled sole.

The experiments conducted by us showed that alloys can be obtained by sintering in a high vacuum, with high temperatures, or heat pressing.

For high-temperature sintering in a vacuum, the

briquets should be pressed without a binding - plasticizer, in order not to introduce impurities and to facilitate the vacuum treatment. Optimum pressures for pressing are 7.5 tons/square centimeter in a volume 60-95 by weight % TiC and 5 tons/square centimeter with 5-40 by weight % TiC.

Optimum methods of sintering briquets of prismatic form 10x13x10 or 13x13x10 millimeters in a vacuum 10^{-4}-10^{-5} millimeters rt. st. by induction heating are shown in Table 1.

With more than 80% contents of titanium carbide, the temperature and sintering time must be increased still further.

Table 1

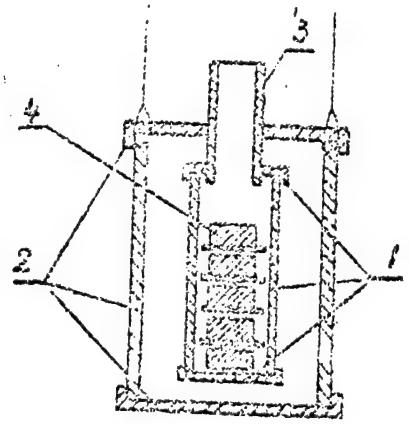
Optimum Methods of Sintering Alloys of Titanium Carbides with Molybdenum

Content TiC wt. %	t° C	Exposure Time Hours	Porosity, % Before Sintering	After Sintering
0-15	2050-2100	2	38	3-7
15-50	2200	2	32-36	3-7
60-80	2300	1	32	7-10

The sintering took place in containers made of molybdenum plate (they were the heaters). Construction of the heating arrangement in shown in the illustration below. During heating, each of the forms is placed separately on a plate made out of tantalic plate. Maximum losses in

weight(in %) during sintering, as a result of vaporization of molybdenum in the vacuum (exposure time 45 minutes) are as follows: 0; 0.5; 1.0; 2.5 at corresponding temperatures 1500, 1800, 2050, 2200 degrees.

The optimum method of heat pressing for all compounds is 80-100 kilograms/square centimeter, 2350°C, during 2-5 minutes exposure



Container-Heater for High-Temperature Vacuum Sintering with Induction Heating

Legend: 1) Heater made out of molybdenum or Tantalic plate;
2) Molybdenum shield with lids having longitudinal section along the generatrix;
3) Inspection tube (molybdenum or tantalum);
4) Forms.

The pressing was accomplished on a laboratory lever press, the construction of which is described in Information Letter No 26 of 1957. Graphite press-forms

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14-16 millimeters were used. The porosity of the pressed forms was within the limits 0-7%.

Even though in heat pressing greater shrinkage is attained than in vacuum sintering, for preparing clean alloys, without impurities, preference must be given to vacuum sintering, since heat pressing leads to noticeable carburization of the forms.

It should be noted that the optimum methods for preparing alloys lie with temperatures, during which the liquid phase already is evident.

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PRODUCTION OF TITANIUM NITRIDE FROM SPONGE TITANIUM

T. T.

/Following is a translation of an article by T.S. Verkhoglyadova in the Russian-language periodical Metallokeramicheskiye Materialy i Metody Ikh Issledovaniya -Informatsionnyye Materialy (Powder Metal Materials and Methods of Investigating Them--Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev 1959, page 45./

Titanium nitride is a hard, high-melting, thermo- and chemically stable compound, which permits its utilization for the preparation of refractory materials in the composition of stable and heat-resistant alloys, of alloys of electro- and radio-technical use, and for powdering molds and crucibles in metallurgy.

In our laboratory, a method has been worked out for producing titanium nitride out of non-conditioned sponge titanium, which is a waste product of production.

Titanium nitride is obtained by the method of direct nitriding of the waste products of sponge with thickness of grains of 2-5 millimeters, at a temperature of

15

1200°, for two hours, in equipment which has been described in the Information Letter of the IMSS, Academy of Sciences, USSR, No 45, for 1957.

Typical chemical compounds of nitride are shown in the following table.

Typical Chemical Analyses of Titanium Nitride

No. of the Experiment	Content, % Ti	N	Sum (Ti + N)
Calculated for TiN	77.4	22.6	100.00
1.....	78.67	20.95	99.62
2.....	78.70	20.30	99.00
3.....	78.60	20.40	99.00
4.....	78.90	21.10	100.00

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A NEW METHOD OF MANUFACTURING RODS MADE OF REFRACTORY COMPOUNDS

/Following is a translation of an article by V.V. Pen'kovskiy and G.V. Samsonov, in the Russian-language periodical Metallokeramicheskiye Materialy i Metody Ikh Issledovaniya -Informatsionnyye Materialy (Powder Metal Materials and Methods of Investigating Them -Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev 1959, pages 50-52./

The production of shaped articles from refractory compounds of the carbide, boride, nitride and silicide type, is acquiring greater technical significance. Such articles, thanks to the high temperature of fusion of the indicated compounds and their durability during heating in air and in aggressive surroundings, find application in the most diverse branches of industry - in chemical machine-construction, energetics, metallurgy, electrotechniques and others /1/.

The most simple articles made out of refractory

compounds are rods with a length up to 1 meter and longer. However, the direct extrusion of rods from the powders of refractory compounds, which is justified in the case of metals [2], is not possible because of the negligible plasticity of carbide, boride, nitride and silicide powders.

In the Institute for metal-ceramics and special alloys, a method was recently worked out for the making of pipes and rods out of refractory compounds [3], consisting of pressing by friction and subsequent extrusion through a spout of the mass made out of the powder of a refractory compound, mixed on a plasticizer (sizing), and of sintering the preparation obtained. However, this method requires preliminary vacuuming of the mass, which complicates the technology of producing the articles both in the laboratories and under industrial conditions. In addition, the raw preparations require long drying before sintering.

Perfection of the method described above consists in pressing of the preparations, without vacuuming of the mass. The use of an alcoholic solution of bakelite, as a plasticizer, makes this possible. As the experiments showed, bakelite is a satisfactory plasticizer.

For making cores from refractory compounds (or mixtures of these compounds), for 100 grams of powder of size 10-50 microns, 25 grams of 45% alcoholic solution

of bakelite are needed; in the case of borides and carbides of boron and silica, 52 grams of a 35% solution are needed. The mixture is pulverized to a moist condition (alcoholic content at the end of the pulverization must comprise 16-20%), is rubbed through a sieve, with dimension of mesh 2 millimeters, and lightly dampened with alcohol, after which it is readied for pressing.

Pressing takes place in a special press-form, which is not differentibted, in principle, from that described in operation /3/. The pressure of pressing does not exceed 5 tons/square centimeter (Table 1).

Table 1

Pressure for Pressing Masses Made of the Powders of Various Refractory Compounds on Bakelite

Refractory Material	B_4C	TiN	ZrB_2	60% ZrB_2 40% B_4C	80% SiC 20% $MoSi_2$
Pressure of pressing, tons sq.cm.	2.5	2.2	1.5	2.5	5

Rods with a diameter of 6-10 millimeters and length 600-1000 millimeters were obtained by the method described, it being known that these lengths are not limiting.

After drying in air for 2-3 hours, the rods obtained

are placed in a drying cabinet at 60° C and dried for two hours with the temperature being raised to 150° to remove the alcohol and the polycondensate of bakelite; the latter somewhat hardens the raw preparation. After drying, the rods are sintered in a Tamman stove in an atmosphere of hydrogen or converted natural gas. For sintering, the rods are placed in graphite combustion boats, in which grooves have been cut, which correspond to the diameter of the rods. The grooves are greased with a mixture of flaky graphite with glycerin to avoid the interaction of the rod material with the combustion boat.

In the stove, the rods are exposed, preliminarily, to a temperature of 500°-600° for 30 minutes, for complete removal of the volatile products of the decomposition of the bakelite. The optimum methods of sintering various materials are shown in Table 2.

In the use of this method, the carburization of the material of the rods at the expense of coking of the bakelite must be taken into consideration: the amount of free carbon in the rods comprises from 2 to 5% by weight. This is not of vital significance in the production of rods from carbides and borides; non the less, for example, in materials containing $MoSi_2$, this amount is quite adequate

for the generation of a noticeable portion of the varying composition phase, of the Novotnyy type phase [4]. To stabilize this phase, the rods, containing $MoSi_2$, are exposed to dusting with aluminum oxide, after sintering at 1500° for 6 hours.

During sintering, shrinkage of the preparations is on the order of 10-20%. Porosity of the bars equals 2-10%, while for bars, based on silicon carbide, for which sintering is more difficult, porosity reaches 18%.

This method is also the principal one used in the production of pipes from refractory compounds.

Table 2

Methods of Sintering Various Refractory Compounds

No p/p [po poryadku; in order]	Material	Temp. °C	Time Min.
1	B_4C	2150	15
2	ZrB_2	2300	10
3	60% ZrB_2 + 40% B_4C	2180	10
4	60% TiB_2 + 40% B_4C	2180	10
5	TiN	2200	10
6	80% SiC + 20% TiN	2180	15
7	60% SiC + 40% $MoSi_2$	2080	15
8	80% SiC + 20% $MoSi_2$	2160	15

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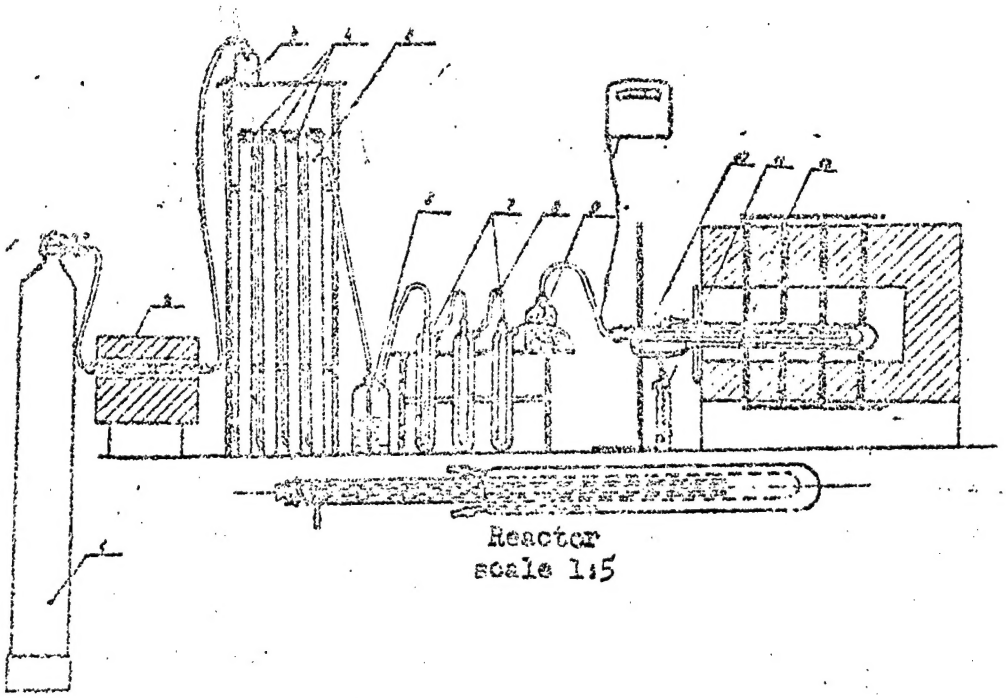
PRODUCTION OF NITRIDES OF REFRACTORY METALS

/Following is a translation of an article by G.V. Samsonov, T.S. Verkhoglyadova, M.M. Antonova and T/V. Dubovik, in the Russian-language periodical Metallokeramicheskiye Materialy i Metody Ikh Issledovaniya - Informatsionnyye Materialy (Powder Metal Materials and Methods of Investigating Them - Reference Material), Publishing House of the Academy of Sciences, Ukrainian SSR, Kiev 1959, pages 53-55./

Nitrides of many refractory metals, including niobium, tantalum, titanium and chromium, are beginning to be used more and more frequently in modern technology, as chemical- and erosion-resistant materials, refractory, and heat-resistant alloys, which well resist abrupt changes in temperature, and also as conducting elements in radio-electronics, in bolometers etc. Technologically, the simplest method of producing pure nitrides is the method of direct nitriding of the powders of refractory metals. This process is somewhat complicated by the necessity of preventing the simultaneous oxidation of the

powders of the metals being nitrided. The nitrogen was passed through a special cleaning system (see diagram) to clean it of oxygen.

Nitrogen from cylinder 1 passes through copper shaving, heated to a temperature of 600-700 in a tubular stove 2 with a porcelain pipe. Then, the nitrogen passes through a system of glass cylinders 4, with diameter 40 and length 900 millimeters, which are filled with copper shaving in a saturated solution of chlorine ammonia in ammonia gas, which serve to absorb the oxygen, and cylinder 5, filled with diluted sulphuric or hydrochloric acid and designated for the neutralization of the ammonia vapors being carried by the passing nitrogen. The process of purifying nitrogen in these solutions can be conducted until the solution in the next to last cylinder remains colorless. During coloring of the solution to blue, it is necessary to completely change the solutions. During neutralization of the ammonia vapors, being carried off from the first cylinders, the acid solution in the last cylinder warms up sharply and ejection of the solution is possible. For this reason, traps 3 and 5 are provided in the system. For drying, the nitrogen is passed through cylinder 7 with concentrated H_2SO_4, after which traces of oxygen are absorbed by the



Reactor
scale 1:5

pyrogallol 8. Further drying of the nitrogen is accomplished by passing it through the cylinders with concentrated sulphuric acid and, further, with calcium chloride or some other strong absorbent.9.

The nitriding process takes place in the quartz reactor 10 with two walls for preventing the falling of the oxygen of the air into the working area of the stove. The reactor is closed by a rubber plug, from the side of the loading opening, through which a quartz pipe enters for passing the nitrogen over the combustion boat with the powder which is being nitrided, and also a pipe with a soldered end for the thermocouple. Nitrogen, coming out of the working area of the stove, across the quartz connecting pipe, is directed into the external vessel, from which it goes, through a rubber hose, into the Tishchenko flask with water, which serves as a water solution to form, in the system, excess pressure and the prevention of the falling of air into the reactor through the entry opening.

Pressure of nitrogen in the reactor is maintained constantly, during nitriding, and is regulated by a reducer, during bubbling of the nitrogen, at the outlet in the Tishchenko flask. The temperature of the process is measured by a platinum-platinum-rhodium thermocouple and is

regulated by an electronic potentiometer.

As a result of the research conducted, optimum methods were established for producing nitrides of titanium, tantalum, niobium, and chromium, with their typical chemical analyses, as shown in the following table:

Methods of Producing Nitrides and Their Typical Chemical Composition

Nitride	Temp. of Nitriding, °C	Exposure, Min.	Chemical Composition, % Metal		Nitrogen		Sum Me+N prod.
			Calculated	Produced	Calc.	Prod.	
TiN	1200	60	77.4	77.9	22.6	21.2	99.1
NbN	1200	120	86.9	86.9	13.1	13.1	100
TaN	1200	90	92.81	92.6	7.19	7.3	99.9
Cr_2N	1100	90	88.2	87.6	11.2	11.3	98.9

In the interval of temperature, 900-1050°, a mixture of the nitrides Cr_2N and CrN is produced, it being known that, with an increase of temperature, Cr_2N (of a grey color with a metallic glitter) becomes dominant.

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